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The purpose of this AASERT Award, a companion award to the AFOSR research grants "Mechanics, Transport Properties and Statistical Physics of Granular Media and Geomaterials" (AFOSR F49620-92-J-0037 / 01 Oct. 1991 - 31 Sept. 1994), and "Quasi-static Mechanics and Material Stability of Particulate Media" (AFOSR F49620-96-1-0246 / 01 June 1996 - 31 May 1998), was to provide AFOSR/AASERT Fellowship participation in the development of a non-invasive 3-D experimental visualization technique to probe the microstructure in deforming granular assemblages and particulate suspensions. The technique is based on the generation of transient streaks of colored particles in transparent fluid-particle systems by means of the photochromic effect. Such streaks would then serve as a tracers of motion within the particulate system. As one candidate system, a model transparent "sand" was made from crushed photochomic glass dispersed in a refractive-index matched ZnCl solution. While the speed of the photochromic effect prevented the penetration of a mechanically non-invasive light beam into the system, other means have been explored for introduction of photochromically darkened particles.

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NON-INVASIVE PHOTOCHROMIC-TRACER STUDIES OF PARTICULATE SUSPENSIONS AND GRANULAR MEDIA

submitted to

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Particulate Mechanics and Shock Physics Program
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01 August 1997

ABSTRACT

The purpose of this AASERT Award, a companion award to the AFOSR research grants "Mechanics, Transport Properties and Statistical Physics of Granular Media and Geomaterials" (AFOSR F49620-92-J-0037 / 01 Oct. 1991 - 31 Sept. 1994), and "Quasi-static Mechanics and Material Stability of Particulate Media" (AFOSR F49620-96-1-0246 / 01 June 1996 - 31 May 1998), was to provide AFOSR/AASERT Fellowship participation in the development of a non-invasive 3-D experimental visualization technique to probe the microstructure in deforming granular assemblages and particulate suspensions. The technique is based on the generation of transient streaks of colored particles in transparent fluid-particle systems by means of the photochromic effect. Such streaks would then serve as a tracers of motion within the particulate system. As one candidate system, a model transparent "sand" was made from crushed photochomic glass dispersed in a refractive-index matched ZnCl solution. While the speed of the photochromic effect prevented the penetration of a mechanically non-invasive light beam into the system, other means have been explored for introduction of photochromically darkened particles.

SUMMARY OF TECHNICAL PROGRESS

A copy of the technical part of the original proposal is included below as Appendix 1. The task statement from the original proposal is as follows:

(1) synthesis of photochromic-dye doped particulates and index-matched interstitial liquids,

(2) development of an optical system, for collimating, splitting, and deploying one or more UV beams through the particulate assemblage, and

(3) development of video acquisition and image analysis to interpret dye-streak images in terms of granular kinematics.

For reasons explained below, significant progress was made only on Task 1.

Two candidates for photochromic particles were considered:

(a) commerically available acrylic plastic beads impregnated with an organic (spiropyran) photochromic dye and

(b) commerically available photochromic (silver-halide) glass.

Initial attempts to impregnate commercially available acrylic beads with an organic photochromic dye (a spiropyran) were not successful, suggesting that synthesis of dye-containing beads may be necessary. Subsequent efforts were devoted to (b), since photochromic glass appeared much more promising in the short term.

We have been able to synthesize a highly transparent "photochromic sand" consisting of crushed photochrornic glass (provided by the Corning Company) in aqueous ZnCl solutions. The glass (the type employed in sunglasses, automotive sunscreens, etc.) darkens on exposure to UV light and remains in a dark-colored state for several minutes. Unfortunately, the darkening is rapid and impedes deep penetration of the excitational UV beam. Hence, short of a major effort to synthesis a new kind of photochromic glass with different kinetics of darkening, the use of mechanically non-invasive UV beams seems to be ruled out at present. We therefore have explored two other techniques:

(i) introduction of UV light by a slowly retracted optical fibers, and

(ii) direct injection of a streak of photochromically colored particles by means of a syringe like device.

While the first method works to a degree, we have not been able with our existing pulsed UV source to attain sufficient levels UV illumination via an optical fiber bundle to allow for rapid creation of long streaks. Hence, we have resorted to Method (ii), which appears to allow for rather rapid injection of a visible streak of photochromically darkened particles, darkened in direct sunlight or in the UV source, into an otherwise transparent fluid-particle system.

At present, we are developing techniques for loading this kind of transparent fluid-particle system, containing dark streaks of particles and confined in a nearly transparent latex membrane, into a tri-axial test apparatus. This should allow us to detect shear bands by direct visualiztion, which to our knowledge has not been accomplished previously.

Apart from the purely technical difficulties discussed above, which are to be anticipated in this type of exploratory research, the project has been beset with the difficulty of recruiting a highly-qualified and committed Ph.D. candidate to make a sustained effort on the research. This is partly due to general problems with graduate recruiting in the P.I.'s academic department during the period of the grant but also partly to the nature of the project, which may sometimes appear to be chemical engineering research to students in appplied mechanics and vice-versa. Furthermore, given its high-risk nature, it perhaps does not have the broad appeal of more main-stream topics in fluid and solid-mechanics. Because of the personnel problem, it has been necessary to sustain the effort on this research project as a kind of sideline to other, higher priorty efforts, and with funding from sources other than the AASERT Fellowship funds. The summary of personnel involved below serves to convey an idea of the overall effort to keep the project going.

Despite the various difficulties touched on above, the ASSERT Award, while not completely expended, has served as an invaluable catalyst in sustaining the research effort, which itself has achieved a promising stage of development and provided much valuable experience to all involved. The techniques developed to date should prove useful in ongoing studies of mechanical instabilities in fluid-particles systems, which is the subject of continuing research and publication by the P.I.'s research group.

SUMMARY OF PERSONNEL INVOLVED

Following is a brief summary of research personnel other than the P.I. associated with the subject research, with dates of participation and parenthetical notes on their major specific contributions to the work:

Fellowship candidates (U.S. citizens, supported by AASERT Grant):

Ms. Laura Nett , Ph. D. Student , Chemical Engineering, 01 July 1993 to 31 August 1993. (Exploratory work on pyrospyran-doped acrylic beads).

Ms. Maria Guevara, Ph. D. Student , Structural Engineering, 01 September 1993 to 31 December 1993. (Initial work on index of refractive matching between crushed photochromic glass and aqueous zinc chloride solutions).

Elizabeth Kristofetz, Ph. D. Student, Materials Science and Engineering, 01 March 1997 to 31 May 1997. (Development of a method for crushing photochromic glass in sizeable quantities employing ceramic compaction apparatus).

Laboratory Assistants (supoported by sources other than the AASERT Grant):

Mr. Pejman Fani, M.S. Student, Applied Mechanics & Engineering Sciences, 1993-1994. (Set-up and repair of pulsed UV source and initial experiments on photochromic glass).

Mr. Ben King, B.S. Student, Chemical Engineering, 1 June 1996 to 31 December 1997. (Preparation of small samples of graded crushed photochromic glass and mixing with index matched ZnCl.).

Ms. Vanessa Le , B.S. Student, Chemical Engineering (UCSD Mc Nair Scholar), 01 January 1997 to present (Working with Dr. Cantelaube in sample preparation and setup of mechanical test apparatus).

Postdoctoral Research Associate (supoported by sources other than the AASERT Grant):

Dr. Florence Cantelaube, Postdoctoral Research Assistant, 01 September 1996 to 31 August 1997. (Exploratory work on various methods of UV excitation of photochromic glass with pulsed UV source and tracer particle injection. Set up of mechanical test apparatus).

APPENDIX I
TECHNICAL NARRATIVE FROM ORIGINAL AASERT PROPOSAL

NON-INVASIVE, PHOTOCHROMIC-TRACER STUDIES OF PARTICULATE SUSPENSIONS AND GRANULAR MEDIA

Abstract

This AASERT proposal requests additional graduate research assistance on the parent AFOSR award "Mechanics, Transport Properties and Statistical Physics of Granular Media and Geomaterials" (AFOSR F49620-92-J-0037 / 1 Oct. 1991 - 31 Sept. 1994). The purpose is to develop a new, non-invasive 3-D experimental visualization technique to probe the microstructure in deforming granular assemblages and particulate suspensions. The major effort will be devoted to development of a 3-D photochromic tracer method based on UV excitation and digital-image tracking of dyed particles in refractive-index matched interstitial liquids, which represents an extension to three dimensions (3-D) of a technique previously developed in our laboratory for 2-D ("Schneebeli") assemblages made from fiber bundles.

All necessary major equipment is already on hand, and this proposal requests three-year support for two graduate research assistants, one to develop 3-D video-image acquisition and processing techniques, and one to work on synthesis of photochromic solid particulates. One candidate, currently enrolled in graduate studies and supported by a first-year departmental fellowship in the P.I.'s institution, is already working on certain aspects of the problem. A second candidate is to be recruited as part of the Fall 1993 graduate recruitment effort in the P.I.'s home department.

Introduction

The parent research program is directed towards the development of continuum models for granular materials and randomly jointed rock masses, based on micromechanical models and statistical physics concepts such as percolation. At present it involves the efforts of the P.I., one graduate research assistant, and one postdoctoral research associate. To date, the effort has been focused largely on theoretical micromechanical modeling, large-scale numerical simulations of granular assemblages, and uniaxial compression experiments in which the yield stress, strain, and electrical conductivity in granular media are simultaneously monitored. One goal is to show the connection between the evolving contact topology and the macroscopic mechanical and transport properties of granular materials.

As generally recognized by researchers in the field of granular materials and particulate suspensions, there is an ongoing need for non-invasive experimental methods which allow one to probe the fine details of particle motion in systems that are normally opaque to visible light. Thus, some efforts have been made to employ x-ray imaging of sand packs containing radio-opaque marker particles [1], and several recent works report on the NMR imaging of flowing particulate suspensions [2]. The first technique requires careful placement of radio-opaque tracers in a granular assemblage and, hence, the arduous preparation of numerous samples for repeated experiments. While the second method, NMR, allows for selective and non-invasive excitation of localized material regions, which then serve as tracers or material markers, the hardware requirements are quite costly for most researchers at this point in time. The present proposal is aimed at a less costly and simpler procedure based on spatially selective excitation of photochromic dyes in optically transparent, model fluid-particle systems.

Since their introduction into fluid mechanics by Hummel and coworkers in the sixties and seventies, photochromic dyes have gained wider usage as non-invasive tracers [3]. Typically a collimated burst of UV light is used to create a visible, colored streak in a transparent liquid stream containing dissolved, normally uncolored dye. The colored streak persists over periods of seconds before fading and serves to label a material line which moves and deforms with the fluid. Unfortunately, most of the better dye materials exhibit good solubility and long excited (colored) state lifetimes only in organic liquids. However, many of the same dyes, incorporated into transparent solids, show much longer excited-state lifetimes. This fact has been exploited in previous work by the P.I. and coworkers [4,5] to study granular assemblages consisting of chopped optical fibers tipped with a dye-impregnated acrylic resin. By means of an optical mask, a transient "dot" pattern, excited by UV light, is imposed on the end of the fiber bundle, thereby allowing one to follow subsequent deformations of the assemblage by video image analysis.

Preliminary results from the above technique have been reported [4,5], and it has recently occurred to the P.I. that the same idea might be extended to 3-D particle assemblages by incorporating the dye directly into transparent particles, which forms the basis of the current proposal.

Proposal

In the present work, it is proposed to employ a refractive-index matched interstitial fluid to create a nearly transparent assemblage of particles doped with photochromic dye. Several collimated UV beams will then be used to generate various grid patterns of excited streaks in the particulate mass, which can subsequently be employed to visualize the deformation and flow of the granular assemblage, by means of video-image analysis.

The development of a suitable refractive-index matched fluid-particle system appears to pose no great obstacle for the present study, since similar systems have been employed over the years for numerous studies of flow in dense particle beds. Most recently, Leighton and coworkers have made use of density-matched acrylic particles to observe wall-slip layers in dense suspensions[6]. While they rely on minor optical imperfections (bubbles or voids) in the polymeric particles in order to track individual particles, a dye-streak technique of the type proposed here could lead to much easier kinematical observations.

The foregoing proposal represents a new direction for the current research and would involve effort well beyond that anticipated in the parent proposal [5], albeit the essential equipment, including a video camera, a pc-based digital-image processing system, and a high-power pulsed UV source are available in the P.I.'s laboratory.

A successful implementation of the above proposal will require three, somewhat distinct tasks:

(1) synthesis of photochromic-dye doped particulates and index-matched interstitial liquids,

(2) development of an optical system, for collimating, splitting, and deploying one or more UV beams through the particulate assemblage, and

(3) development of video acquisition and image analysis to interpret dye-streak images in terms of granular kinematics.

For Task No. 1, the most promising particle material at this time appear to be an acrylic polymer, of the type previously employed [4,5,6], which appear amenable to synthesis and doping by standard polymer chemistry. However, certain chemical modifications may be necessary to ensure that fluid-particle index matching in the visible spectrum will persist sufficiently in the UV to minimize refractive scattering and decollimation of the pulsed UV excitation beams. This concern will be addressed early in the research, in order to ascertain whether alternative materials, such as polymeric or inorganic glasses, will be required.

In the opinion of the P.I., timely progress of the research will require at least two graduate research assistants, with one having responsibility for Task No. 1, the other for Task No. 3, and with both collaborating in Task No. 2. At present, one student, Ms. Laura Nett, whose c.v. is appended below, is the prime candidate for AASERT support on Task No. 3. Currently supported on a first-year departmental fellowship, she is currently gaining experience with video image acquisition and digital image processing.

It is the intent of the P.I. to recruit a new graduate research assistant to the effort in the Fall of 1993. One possibility is contact with the many promising B.S.-degree candidates routinely recommended by letters from other institutions, mainly chemical engineering departments. Another route would be to recruit a highly qualified M.S. student from the many who make inquiries about admission to the Ph.D. program in the P.I.'s department. Given the strong background in chemistry of the typical chemical engineering student, it should not be difficult to identify a candidate with the basic knowledge or competence in polymer synthesis necessary for a significant effort on Task No. 1 during the first year of graduate study. The P.I. would serve as Ph.D. dissertation supervisor for both students and is committed to providing any additional financial assistance necessary to see them through completion of their Ph.D. requirements.

References

1. Private communication, Soil Mechanics Laboratory, Massachusetts Institute of Technology, 1987.

2. J. Rheology (Papers from Special Society of Rheology Symposium, October 1990, eds. R.L. Powell and S.T. Kim), 35, 719-798, 1991.

3. Weidman, P., "Measurement Techniques in Laboratory Rotating Flows," in Advances in Fluid Mechanics Measurements, Springer-Verlag, 1989.

4. Zhuang, X., A Photochromic-Tracer Method for Analysis of Strain Fields - Application to 2-D Granular Assemblages." M.S. Thesis, University of Southern California, 1990.

5. Goddard, J.D., "Mechanics, Transport Properties and Statistical Physics of Granular Media and Geomaterials," Research Proposal to AFOSR, Civil Engineering Program, Directorate of Aerospace Sciences, 15 February 1991 (and revision of 14 May 1991), AFOSR Proposal 91-NA-077.

6. Krishnan, G.P. and Leighton, D.T., "Flow Visualization of the Wall Slip Layer in a Concentrated Suspension Sheared at Low Reynolds Number," Paper 78g, AIChE Annual Meeting, Miami, FL, 1-6 November 1992.